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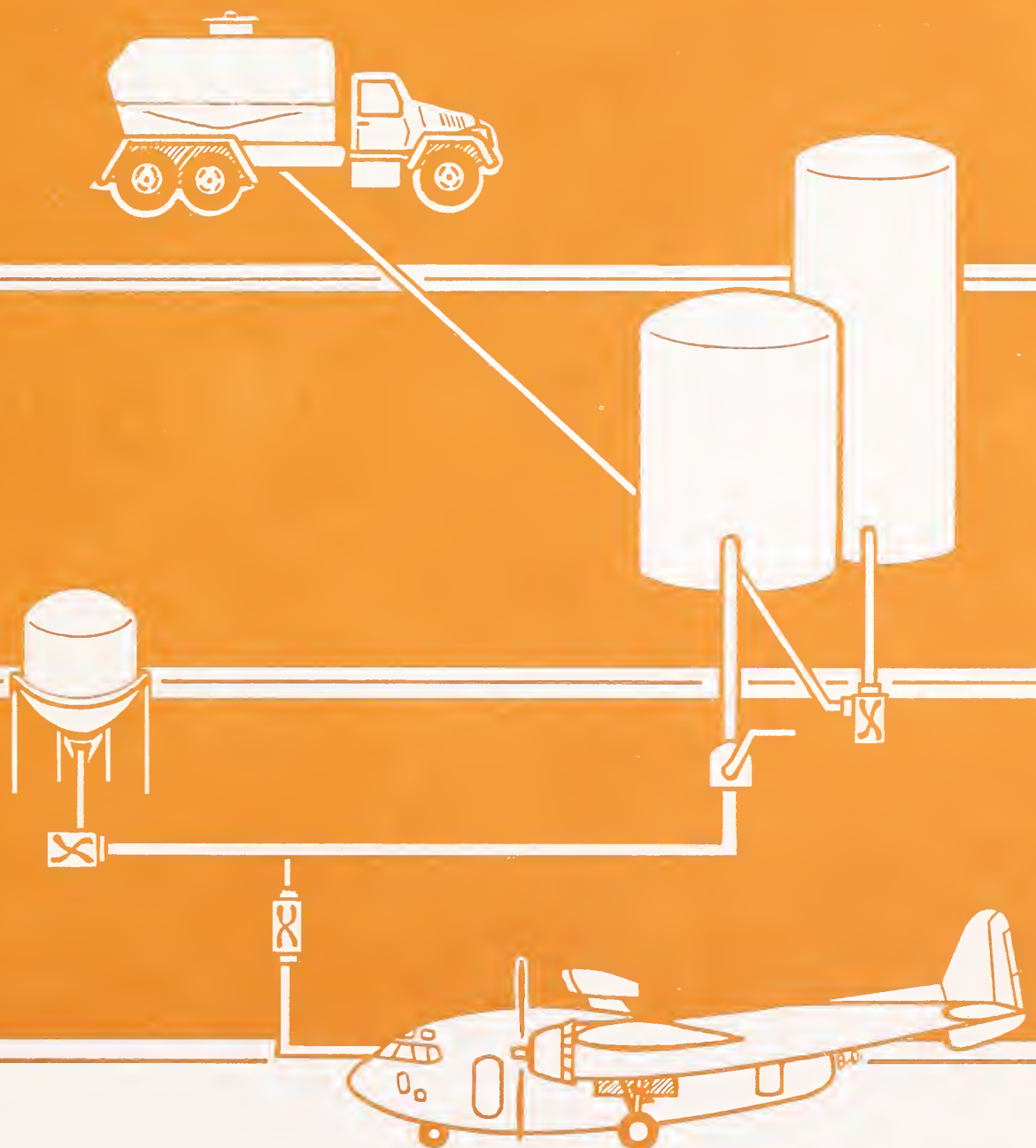
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Evaluation Of Megatard 2700: A Proposed New Fire Retardant System

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Charles W. George and Cecilia W. Johnson



THE AUTHORS

CHARLES W. GEORGE graduated from the University of Montana in 1964 in forest engineering. He received his M.S. degree at the University of Montana in 1969. In 1965, he joined the Intermountain Station's Northern Forest Fire Laboratory staff in Missoula, Mont., where he has conducted studies related to prescribed fire, pyrolysis and combustion, and fire retardant delivery systems. He is currently the leader of the Fire Control Technology research work unit.

CECILIA W. JOHNSON, a chemist in the Fire Control Technology research work unit at the Northern Forest Fire Laboratory, received a degree in chemistry and mathematics from Whitworth College in 1968. She attended graduate school in chemistry at the University of Montana prior to joining the Laboratory in 1971. She works in fire retardant chemical research.

RESEARCH SUMMARY

This report discusses a proposed new fire retardant system and the laboratory analysis and evaluation of the retardant solution produced. The proposed system utilizes ammonium sulfate, delivered dry and liquified at the base, as the active fire retardant salt. For use, the ammonium sulfate liquid concentrate is diluted with water, a gum-thickening agent added, and the final solution delivered to the aircraft as needed. By using alternative thickening packages, the user can have a choice of uncolored or colored retardant.

Attention is given to the physical and chemical characteristics and performance of the final retardant solutions with emphasis on mixing, hydration and viscosity development, solution stability, corrosivity, and effectiveness.

Suggestions supported by performance data are presented for potential product improvements especially in regard to formulation corrosivity. Recommendations are made for additional evaluation including operational testing and a cost-to-benefit (value) analysis.

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Evaluation Of Megatard 2700: A Proposed New Fire Retardant System

BACKGROUND

Omega Chemical Compounding International approached the USDA Forest Service in November 1976 with a proposal to provide a new retardant product utilizing concepts in chemical, storage, and mixing systems different from what was currently in use.¹ Omega thought the new retardant system might fulfill Forest Service needs for a less expensive but effective retardant that could be simply, reliably, and accurately mixed with a minimum of equipment and manpower.

Omega's proposed product was a two-component system combined with water, which produced a thickened product that could be loaded directly into an airtanker. The retardant component contained ammonium sulfate and corrosion inhibitor that would be supplied in dry form by the truckload and liquified upon delivery to the tanker base. The liquified ammonium sulfate, containing the corrosion inhibitor, would then be stored as a 28 percent ammonium sulfate solution. The second component, containing the thickening agent, bactericide, and coloring agent (if desired) would be supplied in 1-ton bulk containers, stored dry, and added to the water stream. The thickened water would be mixed with an equal volume of the liquid ammonium sulfate component as it was loaded into the airtanker. A simplified schematic of the system is shown in figure 1.

This system had a potential for reducing costs because ammonium sulfate costs less than half as much as diammonium phosphate.²

¹Letter to N. Anderson, USDA Forest Service, from L. E. Beightol, Omega Chemical, dated November 22, 1976, containing a "Proposal to USDA Forest Service - submitted by Omega Chemical Compounding International, Glendale, Arizona," 11 pages on file at the Northern Forest Fire Laboratory, Missoula, Mont.

The first step in evaluating the system was to determine the suitability and performance of the final chemical retardant product. Although operational performance depends in part on hardware performance (mixing accuracy and reliability), the components when prepared and mixed under ideal conditions must perform adequately. The Northern Forest Fire Laboratory undertook a cooperative research and development program with Omega to determine certain performance characteristics and to provide data necessary for evaluation.³

The components and the retardant produced by the proposed Omega system do not fit either existing Forest Service retardant specification (liquid, unthickened, or dry, thickened) (USDA Forest Service 1975, 1977). The primary deviations are:

1. One component is stored as a liquid and one is stored dry; the mixed product is thickened.
2. The system is designed to provide mixed retardant to the aircraft on demand, with no storage outside of the aircraft. The length of storage varies according to operational considerations.

To determine the suitability of Omega's Megatard 2700 retardant system, studies were conducted to quantify the performance of the separate components and the mixed retardant. The primary study included but was not limited to areas described in present retardant

²"Chemical Marketing Report," June 7, 1978, through June 19, 1979, lists the price of standard grade ammonium sulfate in car lots as \$60/ton, and feed grade diammonium phosphate as \$240/ton.

³Cooperative Proposal and Agreement, Intermountain Forest and Range Experiment Station and Omega Chemical Compounding International, August 16, 1977, on file at Northern Forest Fire Laboratory, Missoula, Mont.

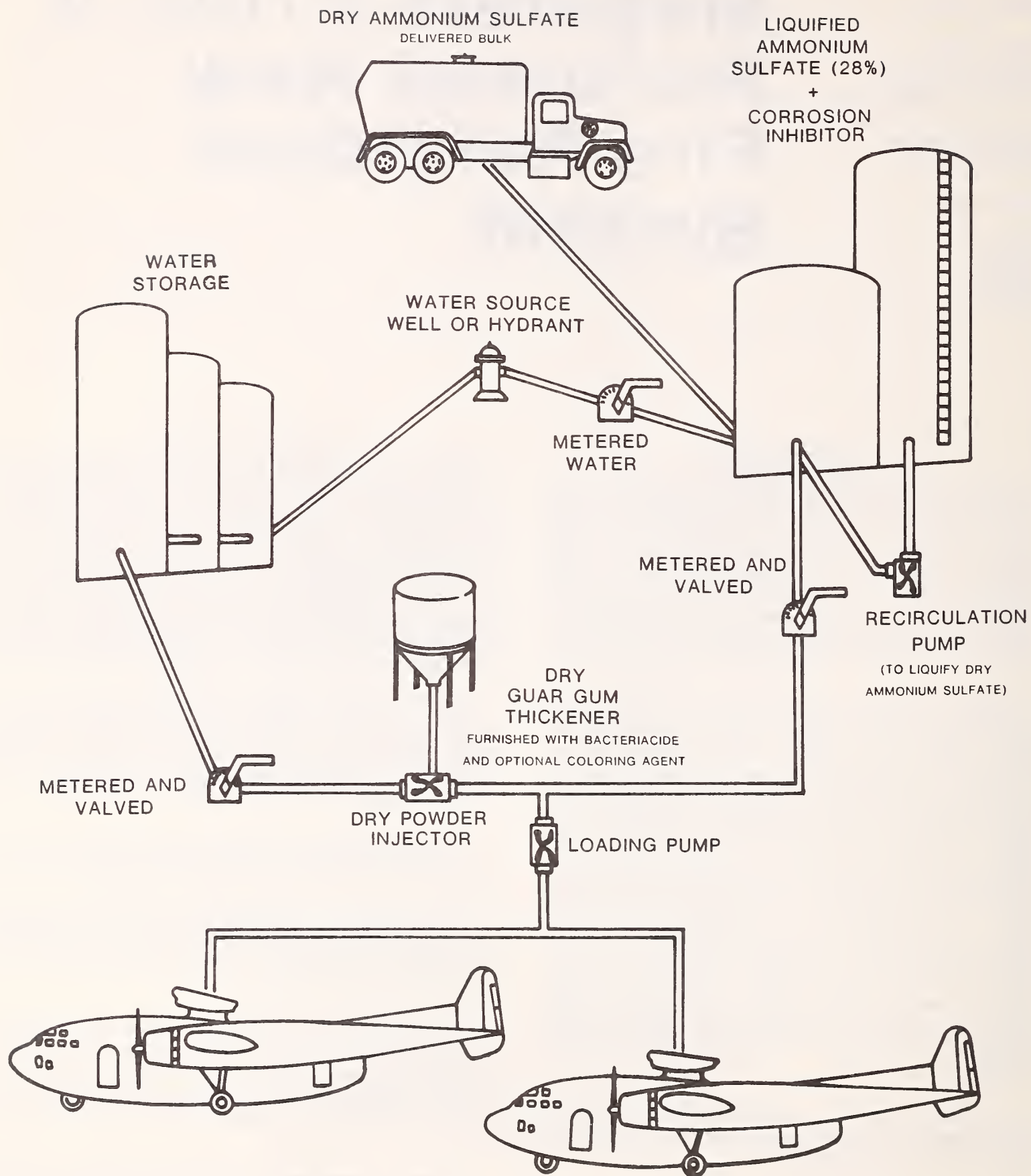


Figure 1.—Simplified schematic of proposed Omega retardant mixing and transfer system.

specifications where specific requirements exist. The performance requirements in the two specifications are not identical due in part to differences in handling and use of the product. The requirements therefore were used only as a guide. A discussion of specific conditions of the proposed methods for handling, storage, and use is included in this paper. Also, the test procedures varied, making it necessary to define performance in a manner not directly relatable to performance required in the specification. In areas where current research programs and results provide additional insight to the performance, a more in-depth discussion is presented.

This report documents the test procedures and results of evaluations performed at the Northern Forest Fire Laboratory in Missoula, Mont.

PRODUCT DESCRIPTION

Two slightly different products, Megatard 2700A and Megatard 2700B, were formulated for the Omega retardant system. Both contain ammonium sulfate as the active fire retardant salt, a guar gum thickener, iron oxide for color, a spoilage inhibitor for the guar gum, and corrosion inhibitors. The only difference between the two products is the type of corrosion inhibitor used: Megatard 2700A contains ammonium thiocyanate and 2700B contains sodium ferrocyanide. The dry component can be supplied with or without the iron oxide coloring agent as specified by the user. A significant change in performance is not anticipated due to removal of the iron oxide. The approximate composition of the formulations and components is given in table 1.

Table 1.—Composition of Megatard formulations and components¹

MEGATARD 2700A	
Liquid component - percent by weight	Dry component - percent by weight
Ammonium sulfate - active retardant, 28 percent	Guar gum-thickener, 78 percent
Ammonium thiocyanate - corrosion inhibitor, 2 percent	Iron oxide - coloring, 18 percent
Water, 70 percent	Spoilage inhibitor, 4 percent
MEGATARD 2700B	
Liquid component - percent by weight	Dry component - percent by weight
Ammonium sulfate - active retardant, 28 percent	Guar gum-thickener, 78 percent
Sodium ferrocyanide - corrosion inhibitor, 2 percent	Iron oxide - coloring, 18 percent
Water, 70 percent	Spoilage inhibitor, 4 percent

¹To mix the Megatard 2700 formulations, the dry component is added to water at a mix ratio of 0.244 lb per gallon of water. The water containing the dry "thickener component" is added in equal volumes to the inhibited 28 percent ammonium sulfate solution (undiluted liquid component).

We received the test sample of Megatard 2700A on June 20, 1977. It consisted of both components in dry form. The ammonium sulfate and corrosion inhibitor were subsequently dissolved in water as would be done by the supplier upon delivery to the base. This provided an opportunity to observe the rate and ease of dissolution of the "liquid component." We received the sample of Megatard 2700B on March 28, 1978. The ammonium sulfate was in liquid component form to be mixed using the same dry component as the earlier sample. (As previously noted, the 2700B liquid component also contained a different corrosion inhibitor.)

We stirred the dry retardant salt/corrosion inhibitor mix for Megatard 2700A into water to form the "liquid component." The ammonium sulfate and inhibitor were easily dispersed and readily soluble with little agitation. The dry component (the color, thickener, and spoilage inhibitor) was mixed with water by simple stirring to form a slurry. This was easily done with rapid dispersal of dry component into the water without clumping or coagulation. Equal volumes of the two solutions were blended immediately to make the "mixed retardant" ready for loading, as would be done operationally.

LABORATORY PERFORMANCE CHARACTERISTICS OF MEGATARD 2700A AND 2700B

The two products were tested for performance using the general procedures and requirements outlined in the USDA Forest Service Specifications, 5100-00301a and 5100-00302b (1975, 1977). The general requirements are given at the beginning of each of the following "Qualification Requirements" sections.

Storage

Dry Chemical Requirement (301): The dry retardant material stored indoors in sacks or bulk for a period of 1 year must be capable of meeting all the requirements listed in 3. The dry retardant, mixed with water according to retardant proportions specified by the manufacturer, shall meet the requirements of 3.3 through 3.16.

Liquid Chemical Requirement (302): The liquid concentrate chemical after being stored in 10-gallon, mild steel containers, outdoors under environmental conditions at Missoula, Mont., and San Dimas, Calif., for a period of 1 year, shall meet all other requirements listed in 3.3 through 3.15. The liquid concentrate mixed with water according to retardant proportions specified by the manufacturer, shall meet the requirements of 3.4 (viscosity), 3.12 (separation), and 3.13 (spoilage) after it has been mixed and then stored as specified in 4.3.1.2 ("mixed retardant") for a period of 4 hours.

Performance: One-year storage tests of Megatard 2700A liquid component and the mixed retardant were initiated in Missoula, Mont., and San Dimas, Calif., as per specified procedures. Throughout the year the test samples were examined visually for evidence of separation or deterioration as well as attack by the retardant on

Table 2.—Characteristics of Megatard 2700A prior to and following storage

MEGATARD 2700A PRIOR TO STORAGE				
Property	Liquid component		Mixed retardant	
Salt content				
$[(\text{NH}_4)_2\text{SO}_4]$				
Kjeldahl analysis	30.65		15.06	
Density (g/cc)	1.171		1.088	
Viscosity (centipoise)	15		1040	
pH	5.5		6.9	
MEGATARD 2700A FOLLOWING 1-YEAR OUTDOOR STORAGE - MISSOULA, MONT.				
Liquid component	Top	Middle	Bottom	Mixed
Salt content				
$[(\text{NH}_4)_2\text{SO}_4]$				
Kjeldahl analysis	27.88	27.75	27.76	28.08
Density (g/cc)	1.169	1.169	1.168	1.169
Viscosity (centipoise)	6	6.5	6.5	5.5
pH	6.87	5.90	7.32	5.34
Mixed retardant				
Salt content				
$[(\text{NH}_4)_2\text{SO}_4]$				
Kjeldahl analysis	10.58	14.30	15.16	14.29
Density (g/cc)	1.065	1.090	1.101	1.090
Viscosity (centipoise)	80	180	710	210
pH	7.31	6.78	6.77	6.70
MEGATARD 2700A FOLLOWING 1-YEAR OUTDOOR STORAGE - SAN DIMAS, CALIF.				
Liquid component	Top	Middle	Bottom	Mixed
Salt content				
$[(\text{NH}_4)_2\text{SO}_4]$				
Kjeldahl analysis	27.88	27.92	28.15	27.87
Density (g/cc)	1.166	1.166	1.167	1.165
Viscosity (centipoise)	8	6	7	7
pH	7.37	7.46	7.50	7.43
Mixed retardant				
Salt content				
$[(\text{NH}_4)_2\text{SO}_4]$				
Kjeldahl analysis	14.39	14.44	14.45	14.45
Density (g/cc)	1.087	1.086	1.093	1.090
Viscosity (centipoise)	41	44	115	55
pH	7.28	8.36	7.84	7.65

the storage container. General appearance was also noted when the storage containers were opened. At the end of 1 year the containers were opened and samples drawn from the top, center, and bottom of each container. All remaining retardant was stirred together. Analyses of Megatard 2700A samples for salt content, density, viscosity, and pH were conducted. The results are shown in table 2. (Other analyses conducted on these samples, such as corrosion, are discussed in later sections of this report.)

A yellow powdery substance that might be part of the inhibitor floated on the surface of the fluid during the year. Some of the fluid also "crawled" out of the can even though the lid had been sealed with a silicone caulking and tightened with bolts. A crust built up to about 1/2-inch thick on the lid by the end of the year.

This phenomenon is characteristic of ammonium sulfate solutions and is commonly observed at Fire-Trol 100 retardant bases. Upon removal of the lid we noticed a strong odor of hydrogen sulfide, likely due to attack of the mild steel by the solution.

The results of chemical and physical tests indicate the liquid component did not change significantly during the year and that it remained uniform throughout the entire container. This is a definite advantage since systematic routine recirculation would not appear necessary. The mild steel storage container, however, showed significant deterioration especially at the liquid/vapor interface and in the vapor zone. About a cup of metal particles (flakes) were lying in the bottom of the can and most of the surface was covered with blisters of metal that had partly flaked off. Figure 2 shows the storage container showing exfoliation and localized corrosion. (The corrosive characteristics of Megatard 2700 will be discussed further under corrosion requirements.)

The mixed retardant showed about 1/2-inch of separation (out of 16 inches total) in 2 months. There was little additional visible separation over the remainder of the year although the solution did appear grainy after 6 months. Complete separation usually follows this grainy appearance, but this had not happened by the end of 1 year. When the 10-gallon storage container was opened, metal flakes floated on about one-third of the retardant surface and the container showed vapor phase corrosion similar to that of the liquid component. Tests on samples of the retardant indicated the top portion was waterlike, while the lowest 2-4 inches were sludgelike and very viscous. Although this bottom portion was thick and viscous, presumably due to settling, no hard particles were found, and slight mixing or agitation created a homogeneous mixture. Results of analysis of these samples are shown in table 2.

The variations throughout the sample may not be significant if Megatard 2700 is used operationally as designed and shown in figure 1, since Megatard 2700 will be demand-mixed and long-term storage is not likely. If storage is required, a short period of recirculation will give a homogeneous solution; although the viscosity may remain somewhat lower than in the fresh mixed retardant.

One-year outdoor storage tests of Megatard 2700B were not performed because the change in formulation (different corrosion inhibitor) is not expected to significantly affect storage characteristics of the mixed retardant. Verification of this would require 1-year indoor storage tests to monitor separation and viscosity. The analysis of the 2700B sample prior to storage is as follows:

Megatard 2700B prior to storage

Property	Liquid component	Mixed retardant
Salt content [% $(\text{NH}_4)_2\text{SO}_4$] Kjeldahl analysis	25.43	15.51
Density (g/cc)	1.159	1.090
Viscosity (centipoise)	7	1,510
pH	6.3	6.9

(The performance of the Megatard 2700, after outside storage, during specific tests will be discussed later.)

Moisture Content

Dry Chemical Requirement (301): The moisture content of the dry retardant material shall not exceed the following values:

Retardant	Moisture content (percent by weight)
Type A Class II (aircraft application, gum-thickened)	3
Type A Class III (aircraft application, clay-thickened)	5

Liquid Chemical Requirement (302): None.

Performance: The moisture contents of the Megatard 2700A dry component (thickener and coloring agent) and the dry ammonium sulfate/inhibitor mixture, which would be liquified upon delivery at the base site under proposed operating procedures, were determined. Samples of the dry components were weighed and placed in a dessicator containing silica gel. After 24 hours the samples were weighed and then returned to the dessicator. Samples were checked regularly until the weight of each sample stabilized. The moisture content of each sample was calculated from the final and initial weights. Moisture contents of three samples were averaged in each case. The moisture content of the dry ammonium sulfate and corrosion inhibitor was 0.01 percent (by weight) and that of the dry component (thickener, color, spoilage inhibitor) was 2.3 percent (by weight). The moisture content of Megatard 2700B was not determined since the small amount of inhibitor in the dry component should not significantly change the moisture content of the salt mixture when inhibitor type is altered.

Specific Weight

Dry and Liquid Chemical Requirement (301, 302): The liquid component and the "mixed retardant" shall be tested at $70^\circ\text{F} \pm 5^\circ$ by weighing a container of known volume before and after filling with retardant. The specific weight of the "mixed retardant" shall not exceed 9.5 pounds per gallon. There is no specific weight requirement for the liquid component.

Performance: All specific weights were determined by filling weighed, calibrated pycnometers with retardant at 70°F and reweighing after entrapped air bubbles had escaped. At least three replications were



Figure 2.—Liquified ammonium sulfate storage container showing exfoliation and localized corrosion.

performed on each sample. The specific weights of the liquid components at the beginning and end of the 1-year storage period were:

	Specific weight	
	Beginning of test period	After 1-year storage
	Lb/gal	Lb/gal
Megatard 2700A liquid component	9.77	9.75
Megatard 2700A mixed retardant	9.07	9.09
Megatard 2700B liquid component	9.67	—
Megatard 2700B mixed retardant	9.09	—

Viscosity and Deterioration

Dry Chemical Requirement (301): The "mixed retardant" when tested using a Brookfield model LVT or LVF viscometer and appropriate spindles shall reach the following viscosity 4 minutes after mixing.

Retardant	Viscosity
	Centipoise
Type A Class II	1000-2000
Type A Class III	1500-2500

A loss in viscosity of more than 25 percent during the first year will constitute unacceptable deterioration.

Liquid Chemical Requirement (302): The liquid component and the "mixed retardant" shall be tested using a Brookfield model LVT or LVF viscometer and appropriate spindles, and the viscosity values before and after the storage period recorded. The viscosity of the "mixed retardant" shall not exceed 200 centipoise. No required viscosity for liquid component. No requirement for deterioration.

Performance: The liquid component and the mixed retardant were tested using the Brookfield model LVF viscometer at the beginning and end of 1 year in

storage. The average values for each sample are given below.

	Viscosity		
	Beginning of test period	After 1-year outdoor storage	Percentage decrease in viscosity
-----Centipoise-----			
Megatard 2700A liquid component	15	6	60
Megatard 2700A mixed retardant	1040	210	80
Megatard 2700B liquid component	7	—	—
Megatard 2700B mixed retardant	1510	—	—

Some concern existed that the large spread in initial viscosities might be caused by differences and non-homogeneity in the dry retardants. The results of further testing, however, suggested that at least some of the differences in the initial viscosities were due to variations in mixing and shear rather than to variations in the products (see discussion on mixing). In common with other gum-thickened retardants, there is a minimum amount of shear required for adequate viscosity development as well as an optimum shear rate to get maximum viscosity and stability from a given quantity of gum.

Additional viscosity tests were run on two 1-quart samples of liquid component and two samples of mixed retardant set aside exclusively for viscosity tests. One sample of the liquid component and one of the mixed retardant contained a mild steel coupon, 1 inch X 1 inch X 1/8 inch (ratio of mild steel to retardant volume in average size operational storage tanks), to determine possible effects of steel on inhibitors or the active chemical. All samples were agitated prior to running the viscosity tests. These tests were run at weekly intervals throughout the 1-year test period. Variations in individual sample viscosities with and without the steel coupon were not significant and the two values were averaged. The results of these tests are shown in figure 3. Note, as previously mentioned, the difference in initial viscosity is possibly due to differences in mixing. Viscosities will be similar if identically mixed. Viscosity can be increased or decreased by varying the use level of the thickener component, as will be discussed later.

Only Megatard 2700A was stored for 1 year as required by the deterioration test specified in the dry chemical, thickened specification. The mixed retardant lost 80 percent of its viscosity by the end of the year. This far exceeds the allowable limit of 25 percent. But since Megatard 2700 was designed for demand mixing and the proposed system does not rely on storage of the mixed retardant, a 1-year storage time may be unnecessary.

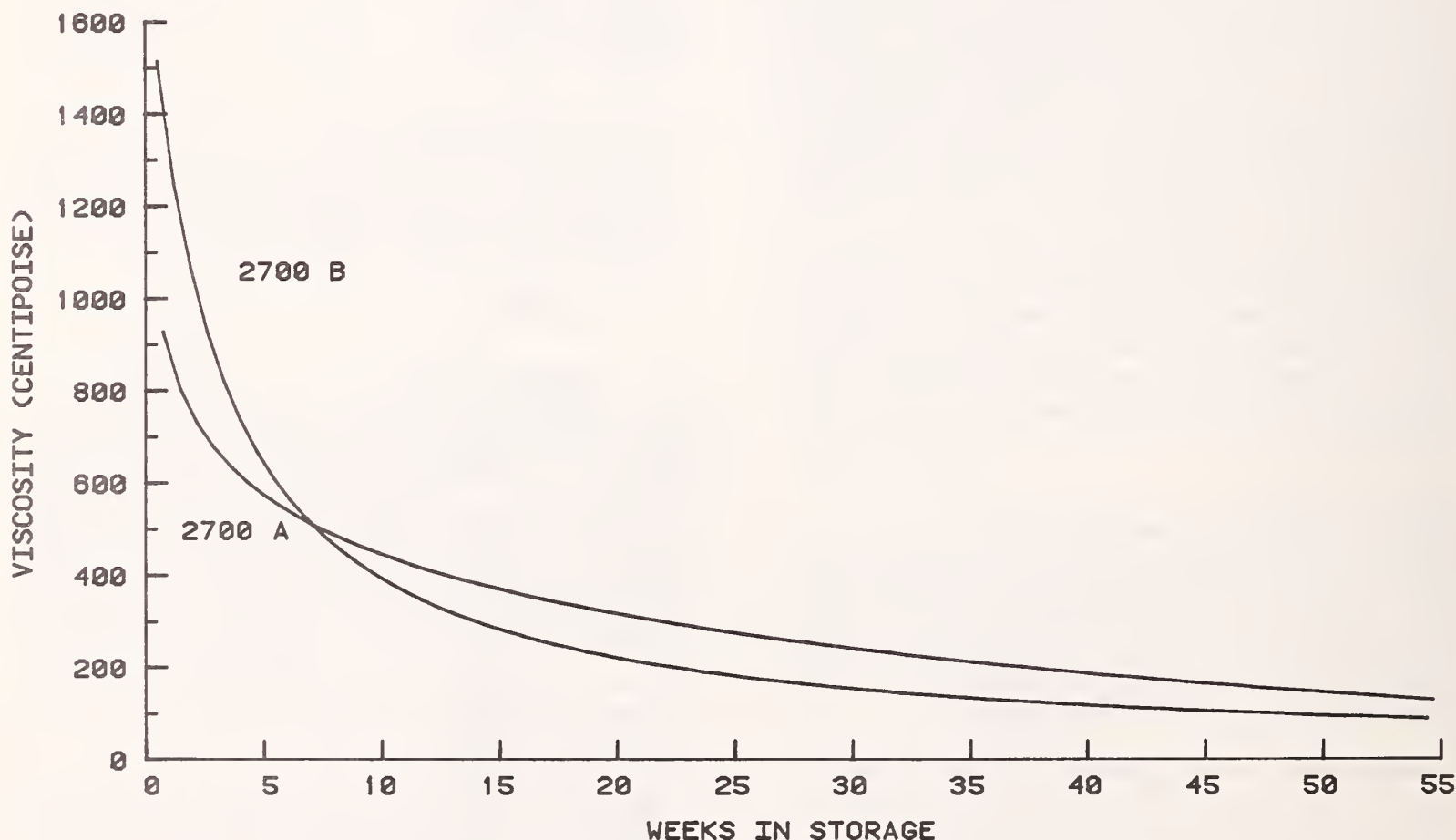


Figure 3.—Viscosity of Megatard 2700A and 2700B during 1-year storage.

A proposed USDA Forest Service specification for all retardants, whether prepared from liquid or dry components, would have reduced the long-term storage requirement to a maximum loss in viscosity of less than 25 percent over a 30-day storage period.⁴ The performances of the Megatard products as per this criteria are given below and shown in figure 4.

	Viscosity		
	Beginning of test period	After 30 days storage	Percentage decrease in viscosity
	-----Centipoise-----		
Megatard 2700A mixed retardant	1870	1640	12
Megatard 2700B mixed retardant	1510	920	39

Although Megatard 2700B had a decrease of nearly 40 percent, it still had a viscosity of almost 1000 centipoise, the minimum allowed for a gum-thickened retardant. (Raising the initial viscosity by altering the mixing procedure to obtain required shear or increasing the concentration of the thickener component can raise the initial viscosity enough that the 30-day viscosity can be increased to greater than 1000 centipoise.)

To illustrate the effect of thickener concentration on retardant viscosity, we varied the amount of dry component used between 0.05 and 0.20 pounds per gallon of mixed slurry while holding mixing time and shear as constant as possible. All samples were stored for 30 days and the viscosities monitored throughout the storage period. Results of these tests are summarized in table 3 and shown in figure 5. Most samples had only a slight change in viscosity during the storage period although the samples with very low concentrations of thickener and thus very low viscosities showed greater instability during storage. In addition, slight changes in gum concentration greatly affected the viscosity. Although there were changes in viscosity during the 30 days, the changes were not significant enough to alter the relationship between the amount of thickener used and the resulting viscosity.

Properly mixed samples of Megatard 2700A and 2700B develop viscosities in the required range (1000-2000 centipoise for gum-thickened retardants) by the time that mixing is finished or within the first 4 minutes after mixing ceases.

Corrosion

Dry and Liquid Chemical Requirement (301, 302): When the liquid concentrate and the "mixed retardant" are tested using the Magna Corrat[™] and 2024-T3 aluminum, AISI C-1010 steel, and naval brass (62 percent Cu, 37 percent Zn, 1 percent Sn) probe tips, the uniform corrosion rate shall not exceed 0.001 inch

Table 3.—Viscosities and percent change in Megatard 2700A viscosity after 30 days storage for formulations prepared using varying amounts of the thickening component

Pound of dry component per gallon of slurry	Viscosity after 60 minutes	Viscosity after 30 days	Percent change in viscosity
-----Centipoise-----			
0.050	110	100	- 9.1
.084	550	150	-72.7
.095	777	220	-71.4
.100	960	890	- 7.3
.107	1180	1090	- 7.6
.122	1870	1640	-12.3
.131	2410	2480	+ 2.9
.143	2970	3400	+14.5
.150	3610	3310	- 8.3
.155	3480	4050	+16.4
.167	4190	4900	+16.9
.179	4760	5000	+ 5.0
.191	5870	6570	+11.9
.200	7040	7080	+ .6

¹Mix ratio recommended by Omega Chemical.

per year (1 mil/yr) and the pitting index shall not exceed 5.0.

Performance: The liquid components and the mixed retardants were tested using standard procedures. The probes were conditioned for 16 hours in the test solution, connected to the Corrat^{er}, and the current flow was monitored for 8 hours in the slowly (1 r/min) moving solution. The probes were removed from the test solution, rinsed under gently running warm water, and allowed to air dry. The probes were then reimmersed and another 8-hour dynamic test monitored. The probes were removed again, rinsed (this time under a very hard stream of cold water), and allowed to air dry. A third 8-hour dynamic test was then monitored. The uniform corrosion rates and pitting index were calculated using a minimum of three replications, all results averaged, and the standard error of the mean calculated. The corrosion rates, pitting indices, and standard errors are summarized in table 4.

The uniform corrosion rate of Megatard 2700A was less than 1 mil/yr on 2024-T3 aluminum and less than 5 mils/yr (moderately corrosive) on 1010 steel and naval brass. Megatard 2700B, which uses a different inhibitor, was subsequently formulated and submitted in an attempt to obtain a retardant salt/inhibitor system that would meet the less than 1 mil/yr uniform corrosion requirement on all three of the specified alloys. This formulation change lowered the corrosion rate on naval brass but nearly tripled the corrosion rate for mild steel. This was unexpected in view of the results of screening tests performed by Ocean City Research Corporation (OCRC) under contract to USDA Forest Service (Gehring 1974, 1978) of several retardant salt/inhibitor combinations (tables 5 through 7). Table 5 shows the results of screening tests utilizing polarization resis-

⁴Proposed USDA Forest Service Interim Specification 5100-00303 for retardant, forest fire, dry or liquid chemical for aircraft or ground application, April 1976.

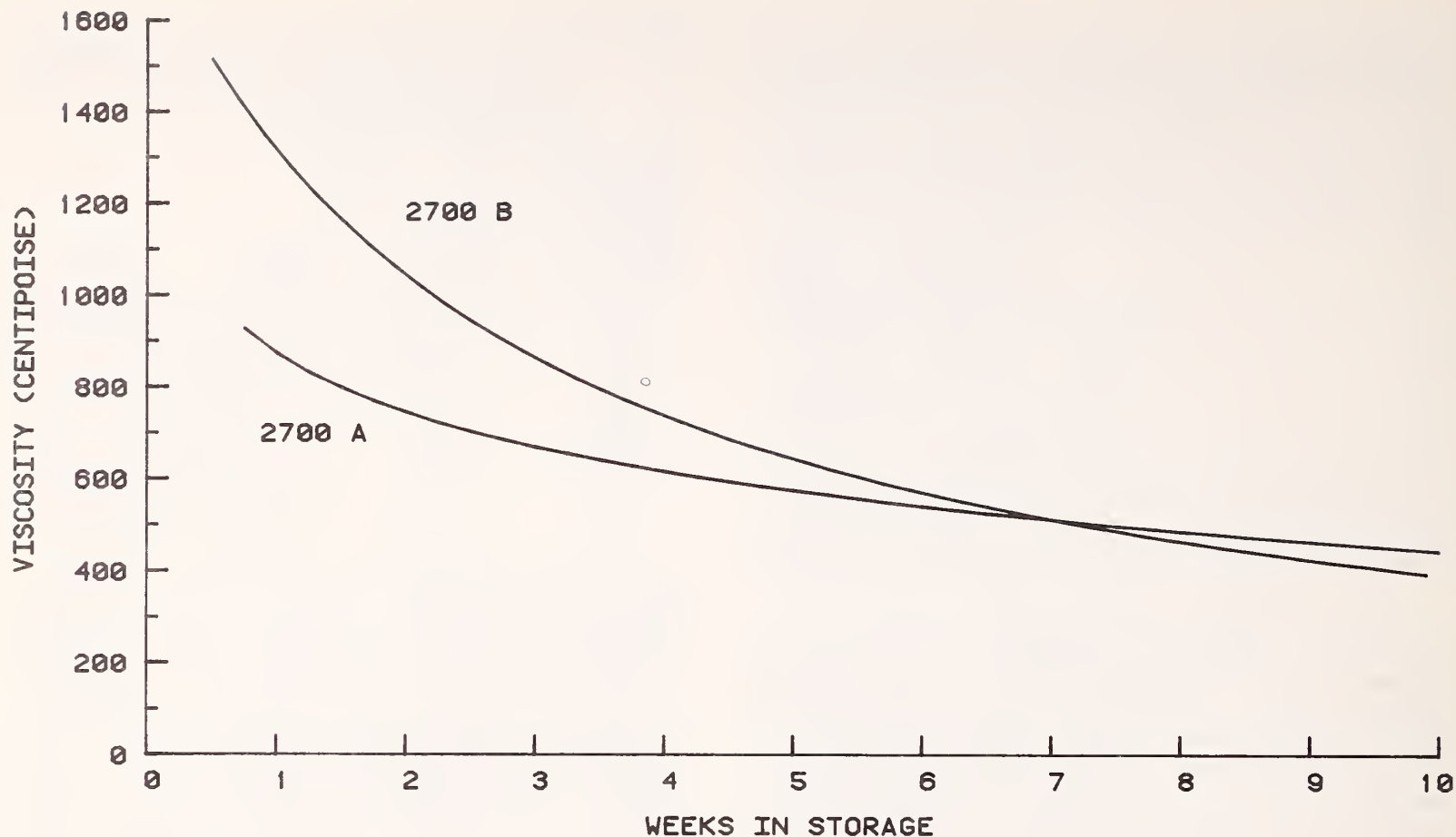


Figure 4.—Viscosity of Megatard 2700A and 2700B during the initial 10-week period (30-day performance shown by a dotted line).

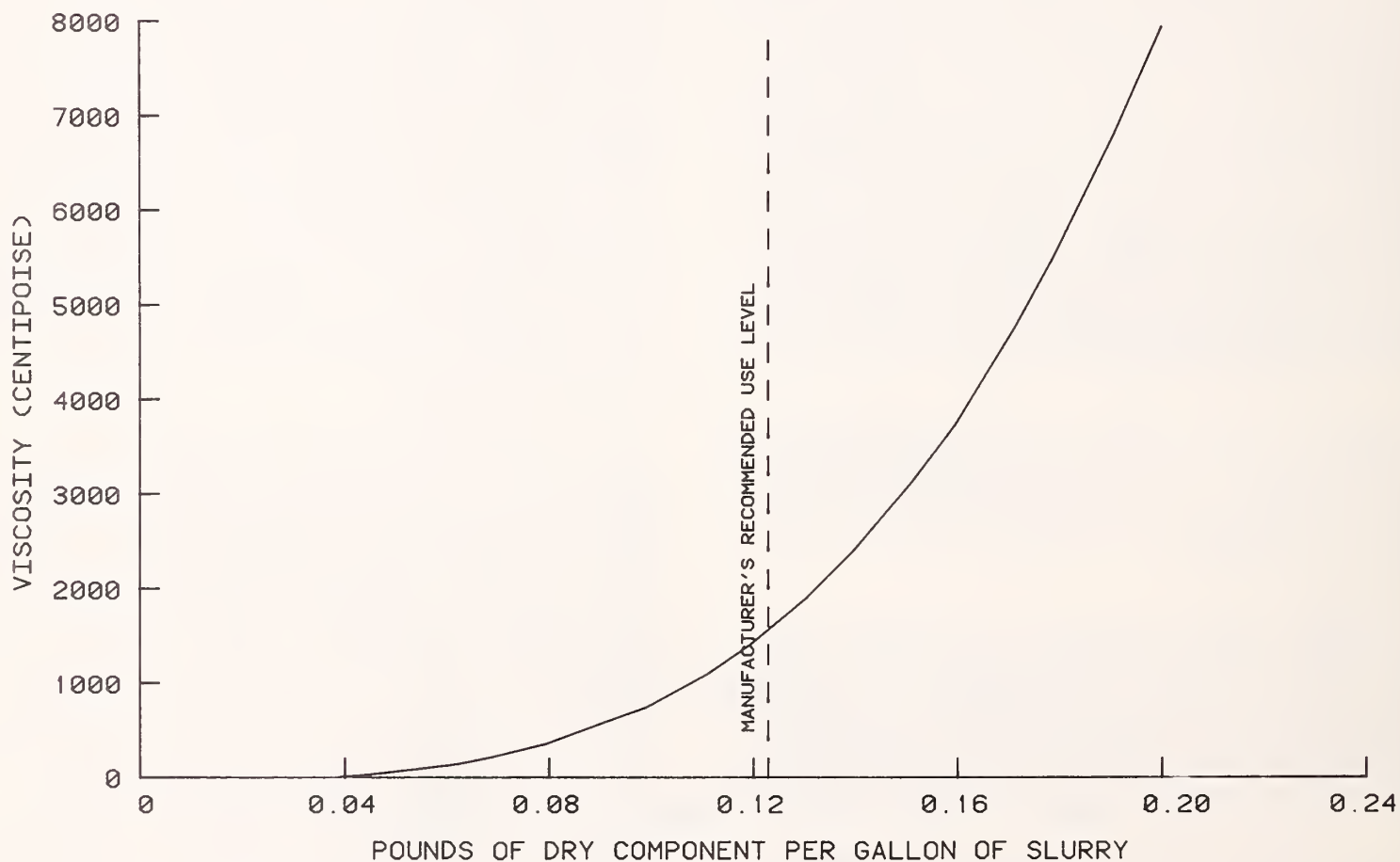


Figure 5.—The effect of changes in thickener concentration on the viscosity of Megatard 2700A.

**Table 4.—Results of uniform corrosion testing of Megatard 2700
using the Magna Corrat**

	2700A		2700B	
	Liquid component	Mixed retardant	Liquid component	Mixed retardant
-----Mils/yr-----				
MEGATARD 2700A AT BEGINNING OF TEST PERIOD				
2024-T3 Aluminum				
Average uniform corrosion	0.44	0.09	0.26	0.21
Standard deviation	.17	.05	.12	.11
Standard error	.08	.03	.07	.06
Pitting index	.31	.06	.15	2.58
1010 steel				
Average uniform corrosion	5.22	3.90	17.50	10.20
Standard deviation	.19	.43	.76	1.16
Standard error	.10	.25	.44	.67
Pitting index	3.12	2.47	17.70	16.60
Naval brass				
Average uniform corrosion	1.03	2.86	.03	.07
Standard deviation	.49	.16	<.001	.06
Standard error	.28	.08	<.001	.03
Pitting index	.17	.73	.03	.03
MEGATARD 2700A FOLLOWING 1-YEAR OUTDOOR STORAGE - MISSOULA, MONT.				
2024-T3 Aluminum				
Average uniform corrosion	0.08	0.16		
Standard deviation	.02	.06		
Standard error	.02	.04		
Pitting index	.07	.05		
1010 steel				
Average uniform corrosion	6.15	.60		
Standard deviation	.45	.05		
Standard error	.31	.03		
Pitting index	2.60	.33		
Naval brass				
Average uniform corrosion	.44	.07		
Standard deviation	.09	<.01		
Standard error	.06	<.01		
Pitting index	.08	.13		
MEGATARD 2700A FOLLOWING 1-YEAR OUTDOOR STORAGE - SAN DIMAS, CALIF.				
2024-T3 Aluminum				
Average uniform corrosion	0.32	0.19		
Standard deviation	.20	.04		
Standard error	.10	.03		
Pitting index	.08	.20		
1010 steel				
Average uniform corrosion	7.29	1.15		
Standard deviation	.30	.04		
Standard error	.20	.03		
Pitting index	3.90	.25		
Naval brass				
Average uniform corrosion	1.85	.46		
Standard deviation	.60	.02		
Standard error	.40	.01		
Pitting index	1.25	.18		

Table 5.—Effectiveness of candidate corrosion inhibitors in ammonium sulfate for common alloys exposed 24 hours (Gehring 1974)¹

Alloy	2024 Al	2024 AlC	6061 Al	7075 Al	Mg	Naval brass	Galv. 1010	4130 steel	304 SS	410 SS
Inhibitor	Corrosion rate (mils/yr)									
Thiourea	0.921	0.921	0.491	0.356		6.17	23.97	2.19	0.0113	0.051
Dimethylamine	.143	.152	.019	.036		1.08	.477	.971	.080	.065
Ammonium thiocyanate	.065	.224	.098	.179	671.7	.167	2.52	.722	.011	.21
Sodium fluorosilicate	1.98	5.87	3.25	1.35		2.08	48.09	46.4	.0073	1.28
Sodium dichromate	1.11	.917	.042	.089		.78	.20	.125	.040	.032
Ammonium fluoride	.484	1.49	.619	4.31		.294	9.04	.437	.0092	.403
Sodium MBT	.161	.143	.028	.035		.216	.271	.306	.018	.098
Sodium nitrite	.424	.0092	.0024	.169		.177	20.7	19.1	.0069	.69
Aniline sulfate	.167	.0402	.033	.067	0.193	1.17	21.95	.655	.0044	.396
Sodium ferrocyanide	.088	.110	.020	.017		.268	.75	.834	.055	.055

¹Determined by polarization resistance measurements at 20 °C.

Table 6.—General corrosion rates for inhibited ammonium sulfate solutions determined by 30-day exposure weight loss and polarization resistance measurements (Gehring 1978)

Alloy		2024 Al	6061 Al	7075 Al	Mg	Brass	4140 steel	1010 steel
Inhibitor		Corrosion rate (mils/yr)						
Ammonium sulfate ¹ without inhibitor	Weight loss ²	0.381	0.32	0.287	—	1.58	3.35	4.17
	initial ³	.017	.028	.024	>250	1.08	1.55	.589
	7 days	.0096	.0092	.0086	—	2.98	2.36	1.31
	14 days	.011	.014	.011	—	2.69	3.51	2.06
	21 days	.0081	.0089	.0087	—	2.09	4.05	3.01
	30 days	.0083	.0080	.0089	—	1.68	3.86	3.71
1% sodium nitrite in ammonium sulfate	Weight loss	.369	.247	.284	—	11.6	10.1	4.09
	initial	.053	.052	.115	>500	24.9	.646	.168
	7 days	.021	.019	.014	—	6.88	.616	.026
	14 days	.0035	.056	.0045	—	15.7	1.82	.085
	21 days	.0039	.166	.0057	—	33.6	9.32	6.66
	30 days	.0036	.348	.0041	—	21.5	9.32	9.11
1% sodium ferrocyanide in ammonium sulfate	Weight loss	.155	.113	.092	—	<.001	.452	.378
	initial	3.65	2.27	2.71	>250	.125	.549	.926
	7 days	.048	.034	.073	—	.059	.652	.775
	14 days	.0098	.0073	.010	—	.053	.664	.870
	21 days	.0056	.0045	.0053	—	.057	1.25	.799
	30 days	.0028	.0029	.0034	—	.080	2.28	.975
1% dimethylamine in ammonium sulfate	Weight loss	.207	.137	.101	—	1.72	2.21	4.28
	initial	.595	.852	1.17	>250	.025	.566	.728
	7 days	.0075	.0082	.0094	—	3.18	.566	1.95
	14 days	.0045	.0065	.008	—	3.10	.657	2.76
	21 days	.0036	.0044	.007	—	3.44	.657	1.76
	30 days	.0036	.0045	.0065	—	3.50	1.87	1.66
1% sodium MBT in ammonium sulfate	Weight loss	.195	.107	.107	—	.814	1.03	2.52
	initial	3.01	1.10	1.64	>500	.051	.361	.561
	7 days	.013	.0089	.017	—	.936	.461	.536
	14 days	.0095	.010	.010	—	1.21	.617	.677
	21 days	.0088	.009	.009	—	1.20	.472	.728
	30 days	.0058	.0065	.008	—	1.15	.510	.765

¹15 percent solution, by weight, 20 °C.

²Corrosion rate determined by weight loss over 30-day period.

³Corrosion rates initially and at 7, 14, 21, and 30 days determined by polarization resistance measurement.

Table 7.—General corrosion rates for sodium ferrocyanide inhibited ammonium sulfate determined under 90-day total and partial immersion conditions (Gehring 1978)

Retardant/inhibitor →		Ammonium sulfate/1% Na ₄ Fe(CN) ₆			
Immersion condition →		Partial		Total	
Alloy		Sample 1	Sample 2	Sample 3	Sample 4
2024 Aluminum	CR (initial) ¹	0.059	0.093	0.037	0.017
	CR (final) ²	.047	—	.043	.059
	CR (weight loss) ³	.076	.085	.065	.132
	Max. pit depth ⁴	1.31	8.61	6—	4.72
Brass	CR (initial)	.258	.618	—	.162
	CR (final)	57.3	43.9	—	.319
	CR (weight loss)	20.2	32.3	—	.102
	Max. pit depth	5—	5—	—	—
4140 steel	CR (initial)	28.0	119	18.7	27.3
	CR (final)	—	82.0	39.7	40.9
	CR (weight loss)	17.1	23.1	11.3	14.0
	Max. pit depth	6.96	14.8	—	—

¹Corrosion rate (MPY) determined initially by polarization resistance measurement.

²Corrosion rate (MPY) determined at 90 days by polarization resistance measurements.

³Corrosion rate (MPY) determined by weight loss after 90 days.

⁴Expressed in milli-inches.

⁵Coupons exhibited ≈ 30-50 percent reduction in thickness at vapor/liquid interface.

⁶Samples with no maximum pit depth given were uniformly corroded over entire surface.

tance measurements after exposure to the solution for 24 hours. Table 6 shows results of weight loss tests for some of the same combinations after 30 days of exposure as well as polarization resistance measurements at 7-day intervals through the 30-day test period. Table 7 shows the results of weight loss and polarization resistance measurements for sodium ferrocyanide in ammonium sulfate over a 90-day interval and includes partial as well as total immersion conditions.

Several factors must be considered when comparing these test results. Corrosion rates vary with the test procedure used, the test conditions (temperature, exposure, etc.), and the length of exposure to the test solution. The results in table 4 were obtained using the Magna Corratrater (linear polarization) during a 3-day time period with 40 hours of actual exposure. During this time the probes were removed from the solution twice and then rinsed and allowed to dry before continuing the test. Depending on the mechanism of inhibition, this procedure might cause a breakdown or fracture of the inhibitor film protecting the probe. At the same time, the movement of the solution over the probes might cause the film to form differently than in a still solution, or not at all. The polarization resistance procedure was performed at varying time intervals up to 90 days. During this time the solution was not agitated and probes remained immersed in the test solution for the entire test period. This might allow a more stable inhibitor film to form (especially over the longer time periods), and the film would be less likely to be disturbed once it was formed. Gehring (1978) concluded after performing the tests summarized in tables 5 and 6 that the extremely short duration (24 hours) of the screening test did not

necessarily reflect long-term performance. Weight loss tests were performed at the end of the test period on the same coupons that underwent the polarization resistance tests so that the same factors would apply. In addition, weight loss test results are an average of the actual loss of metal due to electrochemical activity over the entire test period.

Gehring (1978) also utilized 90-day total and partial immersion weight loss tests in investigating the effect of a 1 percent sodium ferrocyanide inhibited ammonium sulfate solution (see table 7). The partial immersion exposure is usually a more severe test condition in that it includes the possibility for increased electrochemical activity at the liquid/vapor interface. Gehring (1974) well documented that this condition typically occurs at air-tanker bases where retardant is mixed and transferred into storage and/or aircraft for use. A retardant can yield total immersion corrosion rates of less than 1 mil/yr while exhibiting high corrosion rates under partial immersion conditions and during operational use. During laboratory partial immersion tests it is not uncommon to have corrosion extending almost through the entire thickness of a coupon while leaving the two halves nearly untouched. This is demonstrated in the data in table 7 for both brass and steel.

After reviewing results of the earlier corrosion tests, several modifications to the formulas were made in an attempt to reduce the corrosion rate on the mild steel and brass alloys used in the evaluation. Since ortho phosphate is commonly used to inhibit corrosion of mild steel in neutral and basic aqueous solutions (Nathan 1973; Uhlig 1971), diammonium phosphate was added

Table 8.—Uniform corrosion rates of Megatard 2700A and 2700B compared to the corrosion rates for several modified Megatard solutions

Sample	Inhibitors	Alloy	Uniform corrosion	Standard deviation	Standard error	Pitting index	
Mils/yr							
	MEGATARD 2700A						
	1% NH ₄ SCN	2024-T3 aluminum	0.09	0.05	0.03	0.05	
		1010 steel	3.90	.43	.25	2.47	
		Naval brass	2.86	.16	.08	.73	
	MEGATARD 2700B						
	1% Na ₄ Fe(CN) ₆	2024-T3 aluminum	.21	.11	.06	2.58	
		1010 steel	10.20	1.16	.67	16.60	
		Naval brass	.07	.06	.03	.03	
	MODIFIED FORMULATIONS OF MEGATARD 2700						
	Sample 1	1% NH ₄ SCN	2024-T3 aluminum	.23	<.01	<.01	.25
		1% (NH ₄) ₂ HPO ₄	1010 steel	1.57	.02	.01	.28
			Naval brass	2.41	.08	.05	.20
	Sample 2	1% NH ₄ SCN	2024-T3 aluminum	.22	<.01	<.01	.45
		2% (NH ₄) ₂ HPO ₄	1010 steel	.74	.07	.05	2.60
			Naval brass	2.00	.22	.16	.15
Sample 3	0.5% NH ₄ SCN	2024-T3 aluminum	.61	.43	.30	.80	
	0.5% Na ₄ Fe(CN) ₆	1010 steel	3.96	.11	.08	2.10	
		Naval brass	.08	<.01	<.01	.07	
Sample 4	1% NH ₄ SCN	2024-T3 aluminum	.21	.10	.05	.96	
	1% Na ₄ Fe(CN) ₆	1010 steel	2.98	.58	.29	.85	
		Naval brass	.23	.05	.03	.08	
Sample 5	1% Na ₄ Fe(CN) ₆	2024-T3 aluminum	.37	.16	.09	.22	
	2% (NH ₄) ₂ HPO ₄	1010 steel	1.47	1.05	.61	7.70	
		Naval brass	.13	<.01	<.01	.10	
Sample 6	0.5% Na ₄ Fe(CN) ₆	2024-T3 aluminum	.51	<.01	<.01	.21	
	2% (NH ₄) ₂ HPO ₄	1010 steel	6.56	2.60	1.30	16.1	
		Naval brass	.209	.02	.01	.69	
Sample 7	0.5% NH ₄ SCN	2024-T3 aluminum	.29	.08	.06	.58	
	0.5% Na ₄ Fe(CN) ₆	1010 steel	1.02	.02	.01	1.20	
	2% (NH ₄) ₂ HPO ₄	Naval brass	.49	.05	.04	.16	

to several Megatard 2700 samples. Sample 1 was formulated to provide a retardant having a salt concentration of 14 percent ammonium sulfate and 1 percent diammonium phosphate in addition to the 1 percent ammonium thiocyanate. Sample 2 was similarly prepared to yield 13 percent ammonium sulfate, 2 percent diammonium phosphate, and 1 percent ammonium thiocyanate. Standard Corraters tests were then run in the manner described earlier. The results were as expected (the results of these and tests to be discussed are given in table 8). The corrosion rates on mild steel were significantly decreased with the addition and increase in concentration of ortho phosphate. These formulation changes also resulted in a slight decrease in the corrosion rate of brass.

In a further attempt to obtain lower corrosion rates for naval brass, a sample was prepared containing a combination of the corrosion inhibitors used in the Megatard 2700A and 2700B formulation (sample 3). In this sample, the final inhibitor concentration was 0.5 percent ammonium thiocyanate and 0.5 percent sodium

ferrocyanide. An additional sample (sample 4) with the same two inhibitors each adjusted to 1 percent was also tested. Both samples yielded a corrosion rate of less than 1 mil/yr on brass. The corrosion rate of 1010 steel, however, was 3.96 and 2.98 for the two samples, significantly higher than the rates obtained when a 1 or 2 percent diammonium phosphate was used as an inhibitor.

Diammonium phosphate at 2 percent was added to Megatard formulations containing 0.5 and 1 percent sodium ferrocyanide and tested (samples 5 and 6). Sample 5 containing 0.5 percent sodium ferrocyanide and 2 percent diammonium phosphate caused only a slight improvement in corrosion inhibition over standard Megatard 2700B. The sample containing 1 percent sodium ferrocyanide and 2 percent diammonium phosphate resulted in a significant improvement. The corrosion rate for steel decreased from 10.2 to 1.47 mils/yr, while the corrosion rates on aluminum and brass increased slightly but were still well below the 1 mil/yr requirement.

Finally, a sample was formulated with 2 percent diammonium phosphate and 0.5 percent ammonium thiocyanate and 0.5 percent sodium ferrocyanide and tested (sample 7). This inhibitor system lowered the corrosion level on mild steel to approximately 1 mil/yr, while maintaining low corrosion rates for aluminum and brass.

The results of these additional tests suggest that a combination of Megatard 2700 inhibitors with diammonium phosphate is preferable to a single inhibitor for the proposed gum-thickened ammonium sulfate system.

New corrosion requirements were proposed in January 1979, for inclusion in USDA Forest Service specifications for forest fire retardants. The proposed test procedure included 90-day weight loss tests under both total and partial immersion conditions at 70° and 120° F. The proposed requirement provided a maximum allowable corrosion rate after 90 days of 2 mils/yr for total immersion conditions and 70° F partial immersion conditions, and 5 mils/yr for 120° F partial immersion conditions. Incorporation of this test will likely occur following verification of the requirements utilizing existing approved retardant formulations. Final requirements will likely be set as to eliminate formulations having higher corrosion rates than those currently approved and in use.

Megatard 2700A and 2700B were tested according to the proposed procedures and the results are shown in table 9. Table 9 also includes the performance of Fire-Trol 100™, which was included in the test for comparison purposes as an approved ammonium sulfate based fire retardant. The results of the total immersion weight loss tests confirmed the ability of the 1 percent solution of ammonium thiocyanate or sodium ferrocyanide to inhibit corrosion to aluminum. Both inhibitors were effective in reducing corrosion to brass and steel; however, at the elevated temperatures the corrosion rate for the sodium ferrocyanide formulation was significantly higher for steel (1.97 mils/yr as compared to 0.56 mil/yr; allowable under the proposed weight loss procedure is 2 mils/yr).

Results of all partial immersion tests gave a less than 1 mil/yr corrosion rate for aluminum. Results for steel and brass were less consistent; both Megatard 2700B and Fire-trol 100 gave corrosion rates of greater than 5 mils/yr for brass (5.4 and 6.1 respectively) while rates for mild steel were between 1 and 4 mils/yr (Fire-Trol 100 corrosion rates falling between those for Megatard 2700A and 2700B; the proposed requirement was 2 mils/yr). At the higher temperature (120° F) all three formulations exceeded 5 mils/yr on steel.

Weight loss tests under all conditions indicated catastrophic corrosion to magnesium when exposed to any of the three formulations.

The advantages of combining inhibitors and/or add-

Table 9.—Results of 90-day weight loss tests performed on Megatard 2700A, Megatard 2700B, and Fire-Trol 100

	Megatard 2700A	Megatard 2700B	Fire-Trol 100
-----Mils/yr -----			
Total - 70° F			
2024-T3 aluminum	<0.001	<0.001	<0.001
4130 steel	.119	.265	.051
Yellow brass	.068	.001	.016
Az-31-B magnesium	>100.	>100.	>100.
Total - 120° F			
2024-T3 aluminum	.184	<.001	.002
4130 steel	.562	1.97	.083
Yellow brass	.133	<.001	.006
Az-31-B magnesium	>100.	>100.	>100.
Partial - 70° F			
2024-T3 aluminum	<.001	<.001	<.001
4130 steel	3.90	1.13	2.65
Yellow brass	.057	5.41	6.14
Az-31-B magnesium	>100.	91.3	51.4
Partial - 120° F			
2024-T3 aluminum	.483	.034	.026
4130 steel	13.2	9.92	6.53
Yellow brass	.259	.083	31.6
Az-31-B magnesium	>100.	>100.	>100.

ing diammonium phosphate were not investigated using any of the weight loss procedures.

We sent the aluminum test coupons used for the weight loss tests on Megatard 2700A and 2700B to Ocean City Research Corporation for sectioning and microscopic examination for intergranular corrosion. None of the coupons showed signs of intergranular corrosion. Limited pitting on several of the coupons occurred, however, when exposed to partial immersion conditions.⁵ This confirms results obtained by the San Dimas Equipment Development Center, which had examined aluminum, brass, and steel Corratel probes for intergranular corrosion after exposure for 8 days to samples of Megatard 2700A taken from both the top and bottom segments of the samples stored outdoors at Missoula (discussed in the section on storage).⁶

Although corrosion fatigue tests were not required nor conducted, related test results are of interest and should be considered. Ocean City Research Corporation (Gehring 1978) conducted fatigue testing that indicated sodium ferrocyanide in ammonium sulfate

⁵Memorandum from George Gehring to Charles George dated September 9, 1979, regarding USDA Forest Service Purchase Order #43-0353-9-698, Metallurgical Inspection Services. Memorandum and accompanying data on file at Northern Forest Fire Laboratory.

⁶Laboratory report by Peabody Testing Services, Division of Magnaflux Corporation, Los Angeles, Calif., to USDA Forest Service Equipment Development Center. Metallurgical data on file at the Northern Forest Fire Laboratory.

solutions increased the fatigue life of 2024-T3 aluminum as compared to uninhibited ammonium sulfate. Additional fatigue studies are under way; however, no fatigue testing has been performed on either the complete Megatard 2700A or 2700B formulation.

pH Value

Dry and Liquid Chemical Requirement (301, 302):

When the liquid concentrate and "mixed retardant" are tested using a full-range pH meter having a sensitivity of at least ± 0.1 pH units, the pH value shall be between 5.5 and 8.0.

Performance: The pH of the Megatard liquid components and "mixed retardants" are within the required range:

	pH values	
	Beginning of test period	After 1-year storage
Megatard 2700A liquid component	5.5	5.3
Megatard 2700A mixed retardant	6.9	6.7
Megatard 2700B liquid component	6.3	—
Megatard 2700B mixed retardant	6.9	—

Combustion Retarding Effectiveness

Dry and Liquid Chemical Requirement (301, 302): When the "mixed retardant" is applied to ponderosa pine needle and aspen excelsior fuel beds, allowed to dry, and the beds burned, the combined reductions in rate of spread and intensity shall yield a superiority factor between 0.6 and 1.0 ($\pm S_m$).

Performance: An actual effectiveness evaluation was not performed since the Megatard 2700 products were formulated with a standard fertilizer grade ammonium sulfate (100 percent water soluble) for which considerable data are available (George and Blakely 1972). The results of the earlier tests have not indicated significant differences in the effectiveness of fertilizer grade ammonium sulfates (as opposed to the significant effect of purity of ammonium polyphosphate fertilizers). We made an analysis of trace elements in the ammonium sulfate provided as a component of the Megatard 2700 formulation. This documented the quality of ammonium sulfate being used throughout the evaluation and for possible future comparisons. The trace elements in Megatard 2700A retardant component (part 1) mixed to 15.6 percent ammonium sulfate (component 1 contains ammonium thiocyanate) were:⁷

Zinc	0.6 ppm	Nickel	0.1 ppm
Iron	4 ppm	Chromium	0.2 ppm
Manganese	1 ppm	Lead	1.1 ppm
Copper	7 ppm	Cadmium	<0.1 ppm
Magnesium	38 ppm	Fluoride	0.93 ppm
Calcium	400 ppm	Chloride	1300 ppm

Table 10.—Summary of factors included in rating combustion retarding effectiveness

	1 GPC ¹	2 GPC
Grams ammonium sulfate/ft ²	6.17	12.34
Reduction in rate of spread (percent)		
Ponderosa pine needles	37	66
Aspen excelsior	70	81
Reduction in rate of weight loss (percent)		
Ponderosa pine needles	37	47
Aspen excelsior	51	60
Superiority factor	0.49 \pm .04	0.64 \pm .03
Overall superiority factor	0.57 \pm .03	

¹GPC is gallons per 100 ft².

Although no burn tests were conducted, a "paper analysis" of the performance levels of ammonium sulfate as a fire retardant was undertaken. Data points were generated, plotted, and compared to standard ammonium sulfate effectiveness curves using equations for reduction in rate of spread, reduction in intensity, and superiority factors previously developed (George and Blakely 1972), and the concentration of ammonium sulfate present in the mixed Megatard samples. A summary of the data is given in table 10 and relationships shown in figures 6 and 7. The effectiveness of the formulations at application rates of 1 and 2 gal/100 ft² (GPC) are shown by the points plotted on the standard curves. All points fell near the standard curve. The overall superiority factor of 0.57 ± 0.03 for a 15 percent ammonium sulfate solution indicates acceptable performance; however, a slight reduction in the salt content would result in inadequate ammonium sulfate to obtain the required performance as per present specifications.

Based on previous evaluations, the effect of the addition of the gum-thickener to the ammonium sulfate is not expected to significantly alter the combustion-retarding effectiveness of the mixed retardant. Alterations in the formulation as were made in the corrosion section would not be expected to significantly alter the effectiveness of the overall product. In fact, substitution of 1 percent diammonium phosphate for 1 percent ammonium sulfate as a corrosion inhibitor should increase the combustion retarding ability of the formulation provided a high grade (white-acid) ammonium phosphate is used.

⁷Analysis performed by A & L Agricultural Laboratories, Memphis, Tenn., 1977. Analysis report on file at the Northern Forest Fire Laboratory.

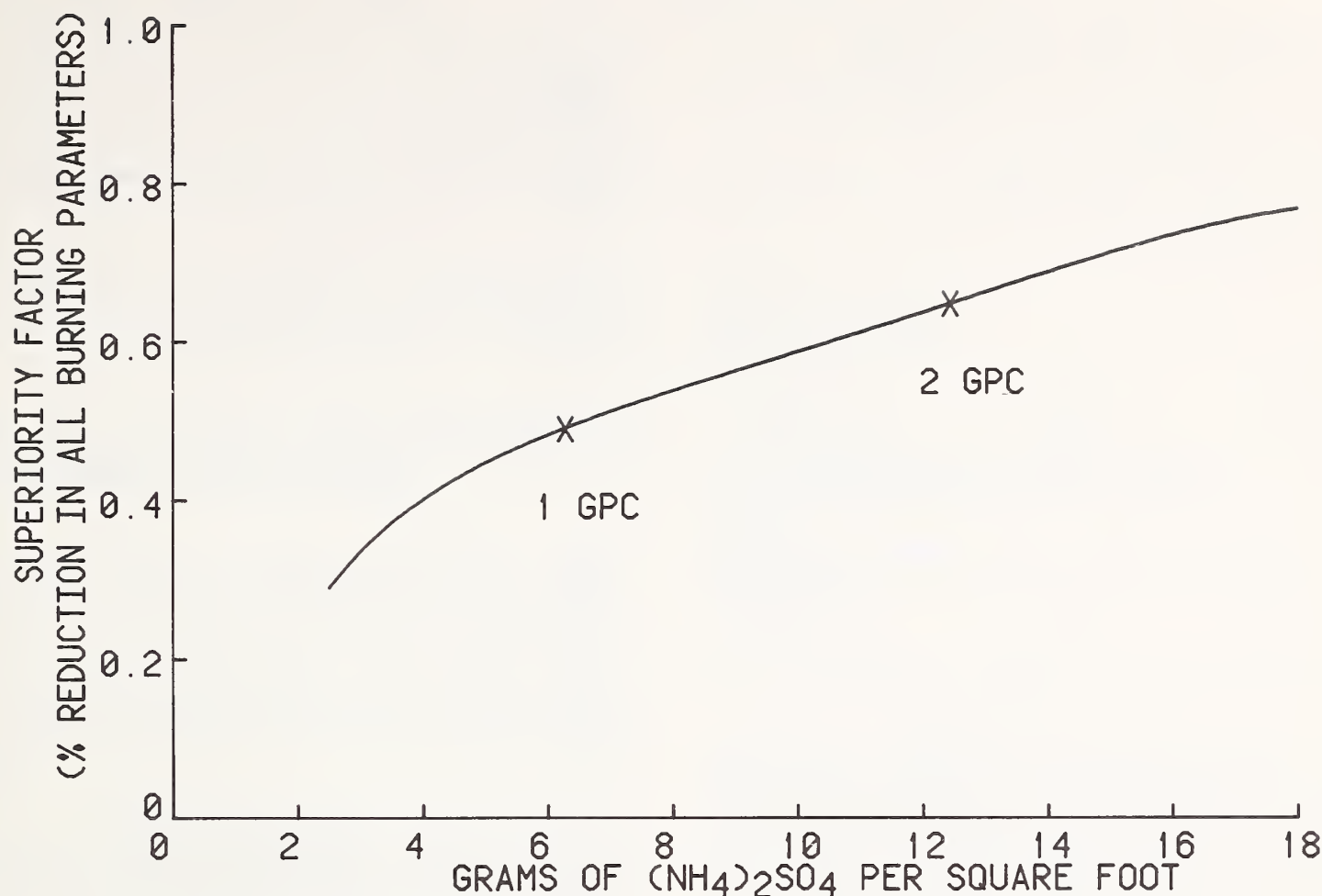


Figure 6.—Estimate of the superiority factor of Megatard 2700 products formulated with ammonium sulfate (AS).

Color

Dry Chemical Requirement (301): The “mixed retardant” shall contain a nonstaining coloring agent in the following proportions of iron oxide, or equal:

Retardant	Gms/gal “mixed retardant”
Type A Class II	4
Type A Class III	10

Dry Chemical Requirement—(Amendment): Visibility when tested. When the “mixed retardant” is dropped onto brush and timber fuel types, experienced observers, in a following light aircraft, shall determine if visibility of the product is acceptable.

Liquid Chemical Requirement (302): Type A liquid concentrate shall contain sufficient nonstaining coloring agent so that the “mixed retardant” shall contain a minimum of 16 grams of coloring agent Fe_2O_3 per mixed gallon or equal.

Liquid Chemical Requirement—(Amendment): Opacity. When the “mixed retardant” is tested in accordance with a specific opacity test (4.3.1.5.1.) the maximum amount of transmitted light shall not exceed an illuminance of 36 lux.

Performance: Properly mixed Megatard 2700 contains 10.25 grams of iron oxide per gallon. This exceeds the limits set for the dry retardants but does not meet

the requirements for liquids. No visibility problems exist using presently approved dry-chemical-based retardants (meeting above color requirements). The level of iron oxide in Megatard 2700 should therefore be adequate.

Amendments to Forest Service Specifications 5100-00301a and 5100-00302b include requirements for visibility and opacity that may be evaluated during testing at San Dimas Equipment Development Center or during an operational evaluation.

Mixing

Dry Chemical Requirement (301): Each retardant will be mixed under specified conditions to determine the retardant expansion and the net horsepower required to mix 1 gallon of retardant.

Liquid Chemical Requirement (302): Mixing shall be limited to pumping proportional volumes of the concentrate and water together.

Performance: The Megatard 2700 system uses components requiring low shear to make a thickened retardant (simple blenders or powder eductors). The dry component is injected into a water stream producing thickened water. The liquified thickener is then pumped together with the liquid ammonium sulfate component forming a thickened slurry that can be loaded directly into an airtanker. No further agitation is required.

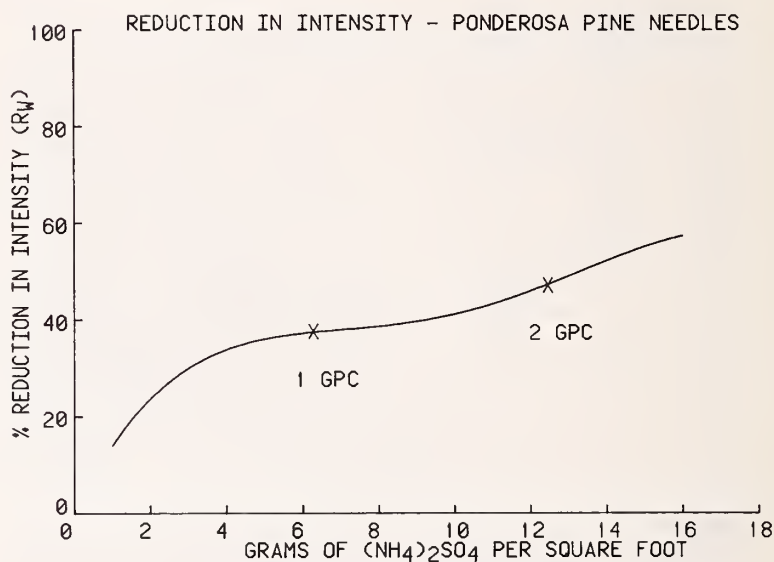
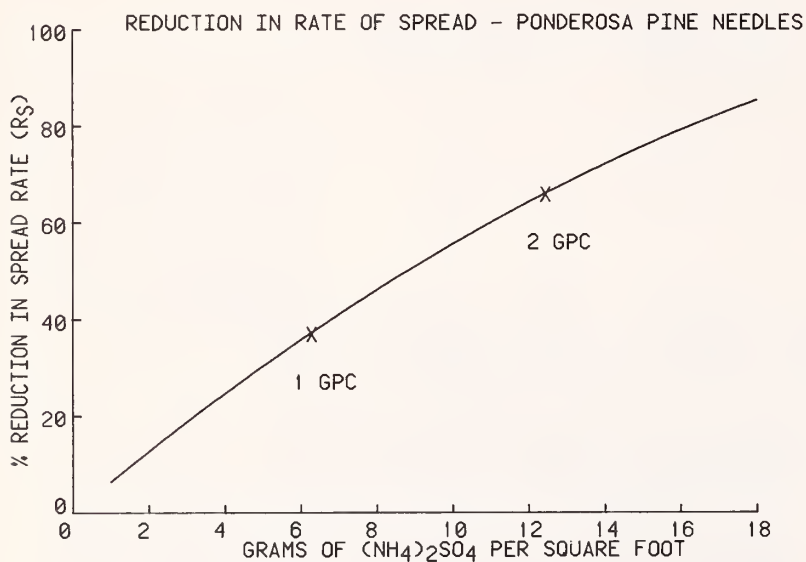
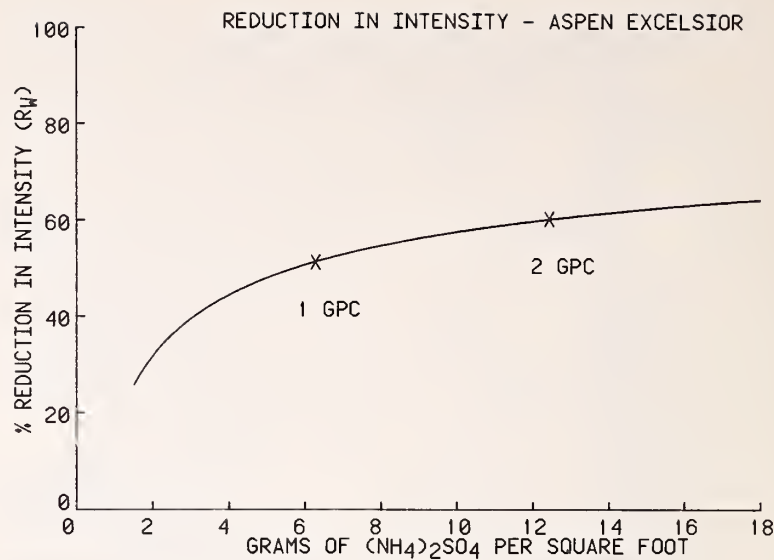
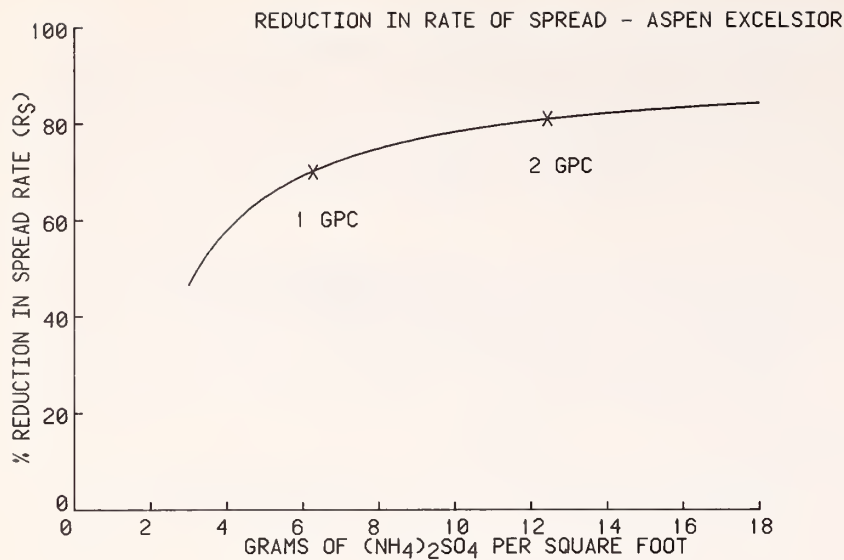


Figure 7.—Relationships used in determining the superiority factor of Megatard 2700 products formulated with ammonium sulfate.

Mixing several batches of Megatard 2700 for use in other tests has suggested that the amount of shear is responsible for much of the variation in viscosity between samples. To confirm this, samples of Megatard 2700 and of Phos-Chek XA (a thickened product that uses a similar gum as the thickening agent) were mixed using a simple laboratory stirrer and a Waring blender for varying amounts of mixing time to alter the amount of shear to the sample. Samples of Megatard 2700A and Phos-Chek XA (a currently approved gum-thickened retardant) were mixed for 3 minutes using the Waring blender, and the viscosities measured at 1, 2, 3, 4, and 5 minutes and at 5-minute intervals thereafter for 60 minutes. Allowing for variations in measurements due to extreme aeration (common in laboratory blended samples) during the first 15 minutes, both samples reached and maintained acceptable viscosities during mixing. Similar results were found when samples were mixed with the laboratory stirrer for 5 minutes. After waiting for 1 hour to allow entrapped air to escape, the viscosities were measured. Results are shown in table 11. Although different amounts of shear were required, generally similar results were obtained with both retardants. There appears to be a definite minimum shear

(which differs with the gum being used) required to obtain adequate viscosity, and an optimum shear to provide the best solution stability. The effect of shear during blending should be a major design input when field equipment is developed. If adequate blending is given originally, the rate of hydration is not a limiting factor; in other words, near maximum viscosities can be attained during mixing and prior to filling the airtanker.

Further testing by San Dimas Equipment Development Center may be conducted if it is necessary to determine the horsepower required to mix a gallon of retardant. The test is normally required for retardants mixed in conventional batch mixers.

Salt Content

Dry Chemical Requirement (301): When chemically analyzed the salt content of the "mixed retardant" will be at least:

Retardant	Percent solution by weight
Type A Class II (gum-thickened)	10.0% $(\text{NH}_4)_2\text{HPO}_4$
Type A Class III (clay-thickened)	15.0% $(\text{NH}_4)_2\text{SO}_4$

Liquid Chemical Requirement (302): When the liquid concentrate is chemically analyzed, the salt content shall not be less than 30 percent P_2O_5 equivalent. After the liquid concentrate has been stored outside for 1 year, the sample shall again be analyzed. The salt content shall be within that obtained before storage by plus or minus 3 percent.

Performance: Megatard 2700 is a dry, gum-thickened, ammonium sulfate-based retardant that does not fit under the liquid chemical requirement or either type or class of dry chemical requirement.

The liquid chemical requirement contains a minimum acceptable salt content for the liquid concentrate of 30 percent P_2O_5 which is not applicable.

Although no requirement for an ammonium sulfate concentrate exists, the 28 percent ammonium sulfate utilized in the Megatard 2700 system is a consideration in determining retardant base storage requirements. There is no technical reason, however, that a gum-thickened ammonium sulfate-based formulation could not meet all performance requirements and operational needs.

The salt content of all Megatard 2700A and 2700B samples was determined by micro Kjeldahl analysis (distillation of ammonia into a boric acid solution and titration of the solution with hydrochloric acid). At least three replicates of each sample were analyzed and results averaged. The results of the chemical analysis indicate that salt content requirements are met:

	Salt content	
	Beginning of test period	After 1-year storage
	$\%(NH_4)_2SO_4$	$\%(NH_4)_2SO_4$
Megatard 2700A		
liquid component	30.65	28.08
Megatard 2700A		
mixed retardant	15.06	14.29
Megatard 2700B		
liquid component	25.43	—
Megatard 2700B		
mixed retardant	15.51	—

Calculation of ammonium sulfate from the determination of ammonia in fire retardants is a common practice; however, the accuracy of the results is dependent upon the purity of the solution. If there is ammonia present that is not in the form of ammonium sulfate ($(NH_4)_2SO_4$), the results will be erroneously high since all available ammonia will be distilled and included in the calculations that are based on all ammonia and all sulfate being tied together in the desired form.

There is a method for the determination of sulfate in clear, nonturbid, dilute solutions. The sulfate reacts with barium to yield a precipitate of barium sulfate. The amount of precipitate can then be determined from the amount of light absorbed during passage through the

Table 11.—Effect of shear on viscosities obtained for Megatard 2700A and Phos-Chek XA

	Viscosity	
	Megatard 2700A	Phos-Chek XA
	-----Centipoise-----	
Mixed using blender: (Waring, low speed)		
Blended for		
0.5 minutes	1010	1800
2.5 minutes	1170	1100
5.0 minutes	1250	840
10.0 minutes	930	410
Mixed using laboratory stirrer: (Precision Scientific, 1750 r/min)		
Blended for		
5.0 minutes	1230	1850

sample. For this method to be applied to fire retardants requires the conversion of a highly colored, opaque slurry to a clear, transparent solution without loss of the sulfate ion. It also requires dilution to the appropriate concentration range, and the performance of the standard analysis precipitation and measurement. As with all multistage determinations the number of possible interferences and inaccuracies increases with the number of steps performed, so that the final result may not be more accurate than the simpler, indirect determination of ammonia.

Separation

Dry and Liquid Chemical Requirement for Aircraft Application (301, 302): When the "mixed retardant" is stored undisturbed in loosely covered glass containers for 1 year, there shall be no visual division of one or more components amounting to more than 5 percent by volume.

If separation of the mixed retardant amounting to more than 5 percent occurs, the separation shall not affect the performance of the retardant. This shall be evaluated by collecting specimens from the top, middle, and bottom one-third of the separated stored products, and retesting against the original requirements as appropriate.

Performance: Two samples of each retardant were placed in loosely covered glass jars. One sample contained a steel coupon, as was discussed in the section on viscosity. All samples were checked daily for the first week and weekly for the remainder of the year. Any separation in the column of retardant was measured, and compared to the total retardant height to obtain percent separation.

The liquid component showed no separation after 4 hours and less than 5 percent separation over the entire

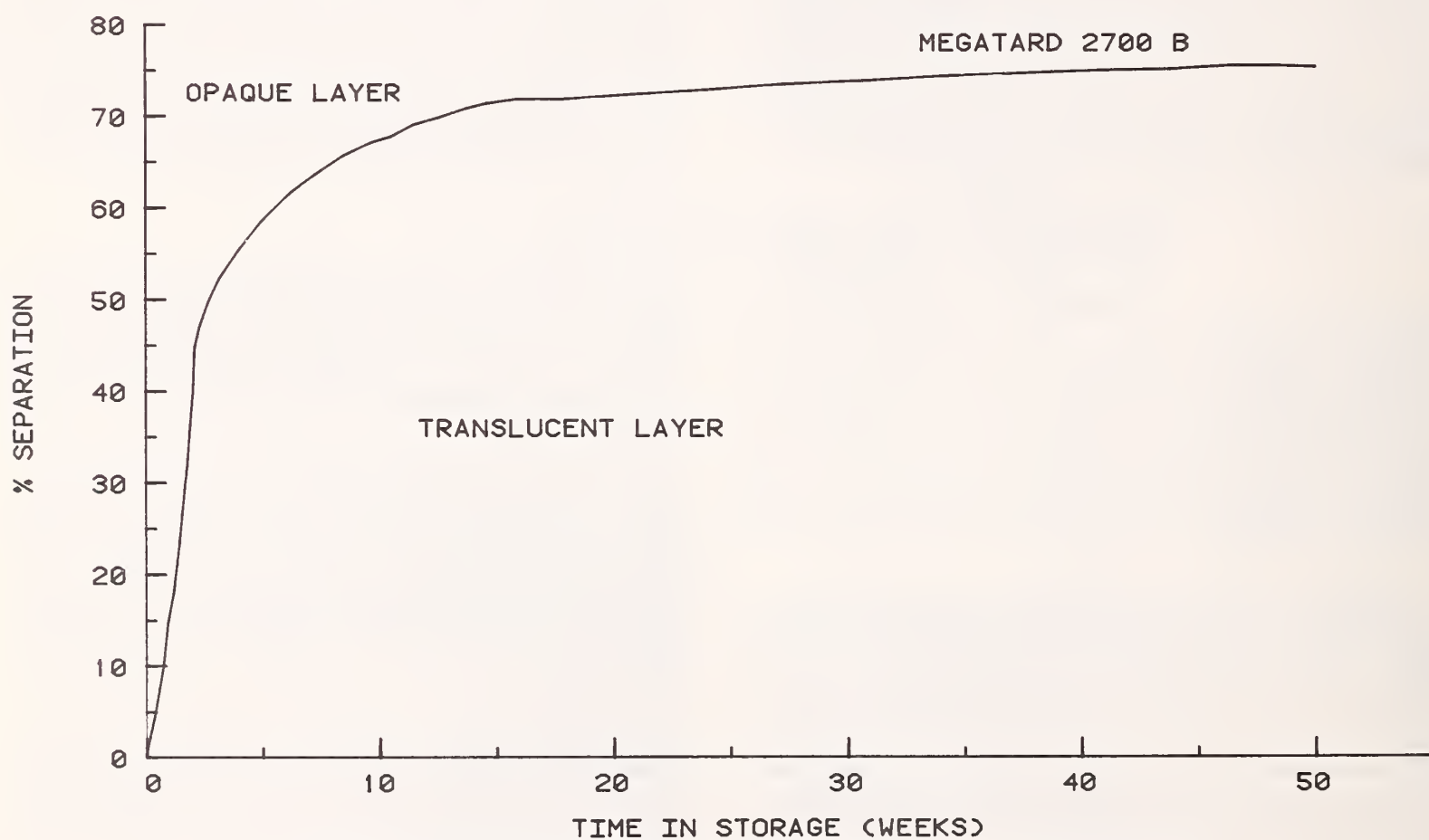
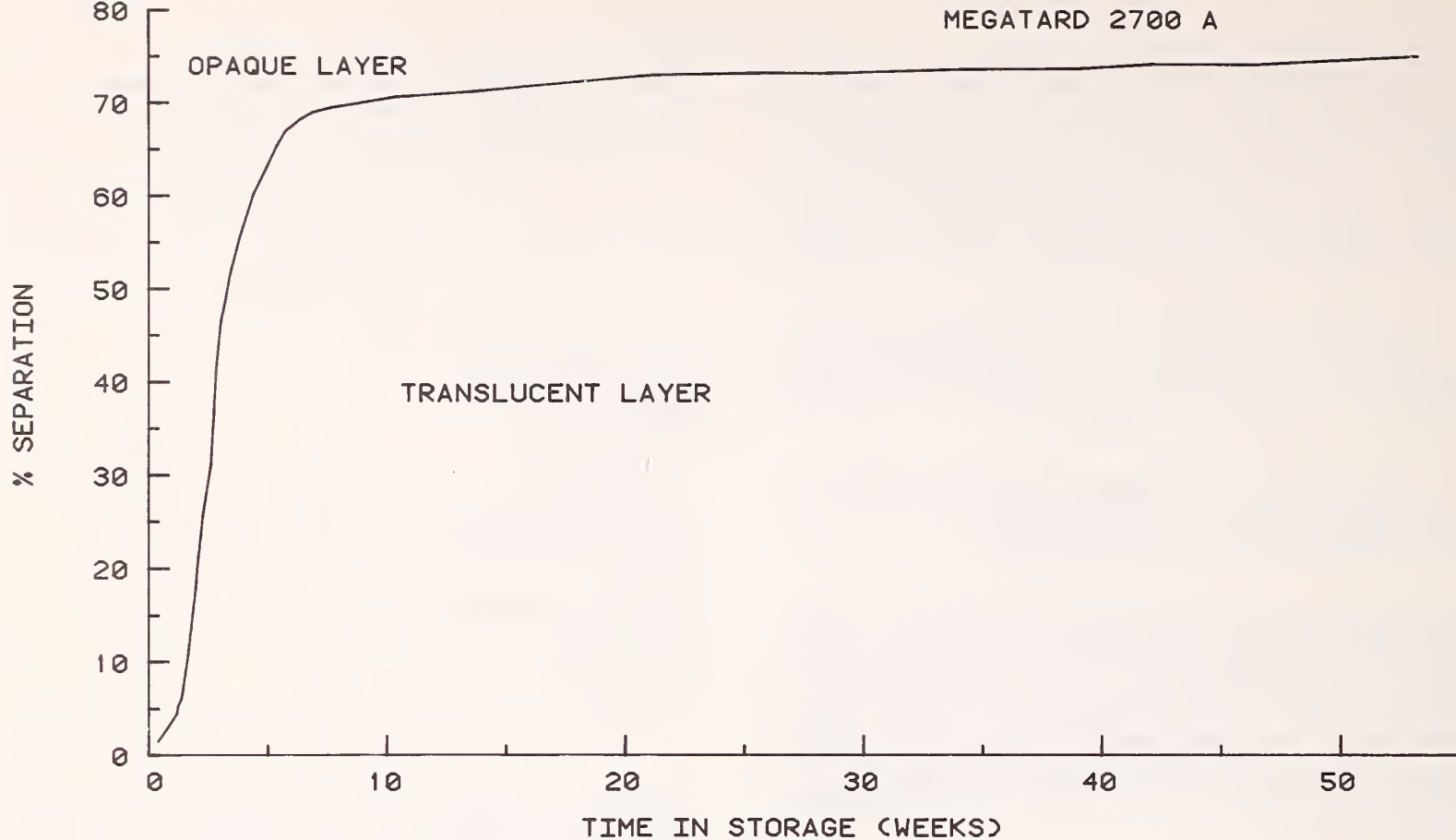


Figure 8.—The effect of storage time on the visual separation of mixed Megatard 2700 retardant.

year. The separation appeared to consist of part of the corrosion inhibitor coming out of solution.

The mixed retardant showed no separation at 4 hours; however, by 30 days about 35 percent separation had taken place in both the 2700A and the 2700B. At the end of 1 year the Megatard 2700A had a very viscous, opaque lower layer, which amounted to about 25 percent of the total volume. A watery, clear, upper layer accounted for 10 percent of the volume, with the center portion (about 65 percent of volume) being translucent but still viscous. After 1 year the Megatard 2700B was about 30 percent separated with a more viscous lower level; the remaining sample graded to a translucent, less viscous material. Figure 8 shows the effect of storage time on separation of Megatard 2700A and 2700B. The performance of the separated components tested is included in the appropriate sections of this report.

Spoilage

Dry and Liquid Chemical Requirement (301, 302): The liquid concentrate and the "mixed retardant" will be placed in glass jars, insulated with 25 ml of *Aerobacter* bacteria, loosely covered, and allowed to stand undisturbed for 3 months. The samples will be visually observed and the viscosity determined during the test period. Marked separation greater than seen in the separation test, a decrease in viscosity, unpleasant odor, or mold growths will be considered as spoilage.

Performance: Preliminary tests were performed to determine the ability of the retardant samples to support bacterial growth. There was no visible sign of bacterial growth nor the musty smell that often accompanies spoilage. Additional spoilage testing may be deemed necessary and performed by San Dimas Equipment Development Center.

Air Drop Characteristics

Dry and Liquid Chemical Requirement (301, 302): At the option of the Government, the aircraft application type "mixed retardant" will be tested for air drop characteristics under standard conditions from B-17 number 22 or similar aircraft. The "mixed retardant" shall demonstrate a capability of producing ground pattern lengths and total recovery equal to or greater than 95 percent of the values produced by the standard retardant (water) as given below:

Drop parameter	Drop height (ft)		
	150	300	375
Total percent recovery	60	55	50
Length of line (length in feet > 1 gal/100 ft ²)	200	175	150

Performance: Drop tests were not conducted since, based on data previously collected (George and Blakely 1973; George 1975), gum-thickened retardants such as Megatard 2700 can unquestionably be expected to

perform better than the standard retardant (water).

An estimate of the drop performance of Megatard 2700 can be obtained by referring to the performance of "gum-thickened" retardants given in the Airtanker Performance Guides⁸ for B-17 tanker number 22.

DISCUSSION AND RECOMMENDATIONS

The performances of two fire retardant formulations, Megatard 2700A and 2700B, were quantified in relation to requirements set forth in USDA Forest Service specifications. Since present specifications are for fire retardants mixed using either all "dry" or all "liquid" components, and since there are inconsistencies in the requirements depending on the method of mixing, handling, use, or for other reasons, no single specification was applicable. Thus, the approach was to consider the requirement for both liquid and dry chemical retardants, the rationale for the requirement, and then attempt to quantify the performance in comparable terms. Also used to provide guidance in the evaluation was a proposed single specification recently developed for either liquid or dry fire retardant (USDA Forest Service Specification 5100-00303, proposed 1977), as well as results of ongoing research programs.

The Megatard 2700 system was proposed as a demand mixed system (retardant transferred from component storage, proportioned, and loaded directly onto the aircraft). One-year storage may not be a necessary requirement if storage of the mixed retardant is not an integral part of the system. In this case, the required storage period should be determined by the need and expected operational storage period (the time between retardant proportioning and use). This time will vary with the fire seasons, areas of use, values, and other factors determining operational requirements. George and others (1977), in a survey of airtanker bases in the six western States in August 1974, indicated 59 percent of the 73 aircraft located at 47 different bases always sit loaded, while 43 percent sit loaded occasionally, and only 5 percent never sit loaded. Although the requirement to sit loaded in some instances is questionable, it occurs in practice for a number of reasons, frequently during periods of high fire danger. The present cost of retardant has already led to requirements such as "airtankers shall land with a retardant load equivalent to their maximum gross landing weight," which will undoubtedly promote the practice of aircraft sitting loaded. The question then is: How long must the mixed retardant be stable? In other words: How long will it hold color, not lose viscosity, or not spoil? Current specifications require 4-hour retardant stability if the mixed retardant is derived from a liquid, and 1 year if derived from a dry product. Separation will not alter the performance of the product in either case. The proposed single specification for retardant from liquid or dry components required a 30-day storage period. This

⁸USDA Forest Service. 1976. Airtanker performance guide for Rosenbaum tank in Evergreen B-17 aircraft (tanker 22). 13 p. USDA For. Serv., Interm. For. and Range Exp. Stn., North. For. Fire Lab., Missoula, Mont.

rationale was used in determining the test matrix to be used in the evaluation and in interpreting the results.

From the results of the tests, no problem is perceived in the long-term storage of the components used in the Megatard 2700 system—that is, either the liquified ammonium sulfate or the dry thickener package. Once the components are mixed the product has fairly good stability; separation occurs but does not significantly affect performance for Megatard 2700A. Viscosity loss over a 30-day period was about 12 percent and at the end of 1 year about 80 percent. Megatard 2700B viscosity loss was higher—39 percent after 30 days storage. As with most gum-thickened products, initial viscosity and rate of development are dependent upon dispersal of the thickener and shear rate during mixing. A gradual loss in viscosity occurred with time; however, no indication of spoilage was observed. A definite performance advantage exists in using Megatard 2700A instead of 2700B in terms of mixed retardant stability (viscosity and separation). The presence of sodium ferrocyanide as a corrosion inhibitor appears to increase the deterioration rate of the gum-thickener although it does slightly reduce the corrosion rate of aluminum and brass when exposed to the solution. Quality of the mixed product can be enhanced by periodic recirculation, although this is not practical after the retardant is mixed and loaded in an aircraft.

One area where the performance of Megatard 2700 is less than desirable is in the area of corrosion. Although Megatard 2700A and Megatard 2700B met present uniform corrosion requirements for aluminum (less than 1 mil/yr using standard Magna Corraters total immersion procedures), the corrosion to mild steel exceeded present 1 mil/yr limits. The average uniform corrosion rate on mild steel exposed to Megatard 2700A was 5.22 mils/yr for the liquid component and 3.90 mils/yr for the mixed retardant, while Megatard 2700B gave a corrosion rate on mild steel of 10.2 mils/yr using the same test procedure and conditions. The corrosion rate for brass exceeded 1 mil/yr for Megatard 2700A, while Megatard 2700B had a corrosion rate well below 1 mil/yr (2.86 mils/yr for Megatard 2700A and 0.07 mil/yr for Megatard 2700B). Results obtained using the standard corrosion test and results of corrosion testing performed by Ocean City Research Corporation under Forest Service contract (Gehring 1974, 1978) led to several additional tests. These were thought to be more meaningful in quantifying corrosion expected during operational use and in testing of some formulation combinations and modifications in hopes of reducing corrosion without significantly affecting other performance characteristics.

Additional testing and formulation modifications demonstrated that if the inhibitors of Megatard 2700A and 2700B were incorporated into a combined formulation and 2 percent diammonium phosphate substituted for 2 percent ammonium sulfate, all present corrosion requirements could be met. Another alternative nearly as good is to use ammonium thiocyanate in combination with 1 to 2 percent diammonium phos-

phate. Increasing the concentration of diammonium phosphate from 1 to 2 percent reduces the uniform corrosion of mild steel and brass. (The corrosion rate on mild steel was below 1 mil/yr; however, the corrosion rate on brass was reduced to only 2 mils/yr.) Test data (table 8) show that the slight addition of sodium ferrocyanide in addition to ammonium thiocyanate and diammonium phosphate will eliminate the brass corrosion problem (reduce brass corrosion rate to about 1 mil/yr).

The fallacy in the 1 mil/yr mild steel requirements and test procedure is that operational experience and research studies conclusively show the primary corrosion problem with mild steel exposed to ammonium sulfate based fire retardant solutions occurs at and above the liquid/vapor interface and not primarily below the liquid surface. Fire-Trol 100, for example, is an inhibited ammonium sulfate based retardant that consistently exhibits total immersion rates of less than 1 mil/yr. Significant mild steel corrosion, however, occurs under field conditions; and laboratory studies show that partial immersion and vapor zone corrosion rates, determined by weight loss measurements after a 90-day exposure at 120° F, are as high as 5 to 15 mils/yr (the proposed requirement is less than 2 mils/yr at 70° F). Existing corrosion data indicate that corrosivity of the Megatard products to mild steel is generally similar to Fire-Trol 100.

Additional testing using total immersion and partial immersion weight loss procedures substantiated these data. Megatard 2700A gave low corrosion rates, less than 1 mil/yr, by weight loss procedure for aluminum, mild steel, and yellow brass under total immersion conditions at both 70° F and 120° F. Megatard 2700B only exceeded 1 mil/yr on mild steel at the 120° F condition (1.97 mils/yr was obtained). Corrosion rates with all products increased with temperature and under partial immersion conditions. Using partial immersion, Megatard 2700A had advantages with yellow brass while Megatard 2700B had an advantage with steel. These advantages are not consistent with the results of total immersion Corraters tests previously discussed.

Further testing could be done to verify that a combination of inhibitors can actually reduce weight loss corrosion under total and partial immersion conditions. However, a combination of the inhibitors of Megatard 2700A and 2700B (ammonium thiocyanate and sodium ferrocyanide) at 0.5 percent, and substitution of 2 percent diammonium phosphate for 2 percent ammonium sulfate, results in corrosion rates of less than 1 mil/yr with all three alloys required in present corrosion specifications and using present test procedures. All formulations and modifications of 2700A and 2700B included in the evaluation met aluminum uniform corrosion requirements, which are the primary concern in aircraft maintenance and safety. Intergranular corrosion of aluminum was not detected on either the exposed Corraters probes or test coupons used in weight loss tests (both total and partial immersion).

Results of corrosion tests at the Northern Forest Fire Laboratory using the Megatard 2700 formulations and by Gehring (1974, 1978) of Ocean City Research Corporation using inhibited ammonium sulfate solutions (without color or thickener), support one of the initial premises and a factor in the design of the test matrix, that is, neither the removal of iron oxide coloring, nor presence of the thickener would have a significant effect on the corrosive properties of the sulfate-based formulation.

The best overall choice (considering only the two original Megatard formulations) is probably 2700A, which provides the lower mild steel corrosion. Brass is primarily exposed to retardant in ground equipment and often can be avoided during hardware selection. Mild steel is more commonly found in ground storage and transfer equipment. A Megatard formulation composed of 0.5 percent ammonium thiocyanate, 0.5 percent sodium ferrocyanide, 2 percent diammonium phosphate (white-acid produced), and 13 percent ammonium sulfate appears to offer advantages over both Megatard 2700A and 2700B. For one, it would not be expected to alter performance to any extent from the Megatard formulations (as per other requirements and test procedures) with the exception of combustion regarding effectiveness, which would be expected to improve if the 2 percent diammonium phosphate were added.

To summarize, two advantages of the Megatard system that should be considered are: (1) gum-thickened products have improved stability and drop performance; and (2) formulations using ammonium sulfate should be considerably cheaper than those using either ammonium phosphate or ammonium polyphosphate. Two disadvantages of the Megatard system are: (1) For the same capability (gallons of mixed retardant) the Megatard system will require more storage capacity (for the 28 percent ammonium sulfate solution) than present formulations. The storage could be reduced considerably if the dry component (ammonium sulfate and corrosion inhibitor package) were stored at the base or in close proximity. (2) Significant corrosion to mild steel is to be expected (similar to that experienced with Fire-Trol 100). It appears, however, that formulation changes can effect a reduction in this characteristic.

We recommend that parameters not quantified in this evaluation per present and proposed specifications be investigated. These should include tests for which San Dimas Equipment Development Center has responsibility; that is, abrasion and erosion, pumpability, color, health, and safety. We recommend that a preliminary "value analysis" be conducted that would consider the trade-offs between performance and reduced cost as a result of the use of ammonium sulfate in relation to requirements stated in present unthickened liquid, thickened dry, or proposed combined specifications. The historic use of similar, currently approved products and their impact on maintenance and replacement

costs should be included. Storage containers of stainless steel, fiberglass, polypropylene, or other inert materials should be considered as an alternative to mild steel. If results of the recommended tests and analysis are positive, an operational system should be evaluated (including full complement of storing, proportioning, transferring, and loading equipment). The evaluation should assess all system variables not measured in the laboratory. The operational evaluation should provide the information needed to complete a cost-benefit analysis.

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This report discusses a proposed new fire retardant system and the laboratory analysis and evaluation of the retardant solution produced. The demand mix system uses an ammonium sulfate based concentrate to which a gum-thickener (with or without coloring) is added as the solution is delivered to the aircraft. Attention is given to the physical and chemical characteristics and performance of the final retardant solution. Suggestions are presented for potential product improvement and recommendations are made for further evaluation and a cost-to-benefit analysis.

KEYWORDS: fire retardant, retardant evaluation, USDA specification, ammonium sulfate, corrosion, hydration, viscosity

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